AIRBORNE ASBESTOS SPECIFIC OPERATIONS CHECKLIST

Instructions to the Assessor: This checklist addresses specific accreditation criteria prescribed in applicable sections of NIST Handbook 150-13.

Place an "X" beside any of the checklist items which represent a deficiency. Place a "C" beside each item on which you are commenting for other reasons. Record the item number and your written deficiency explanations and/or comments on this list or on the comment sheet(s). Place a check beside all other items you observed or verified at the laboratory.

1 Organization and management

1.1 See General Operations Checklist

2 Quality system, audit and review

2.1 The quality system documentation contains the following:

_____ a. a summary outline or Table of Contents that refers to the major portions of the quality manual. If parts of the quality system documentation are kept in different places, then this summary should include the location of the procedures, instructions, records, etc.;

_____ b. descriptions (or reference to descriptions) of the laboratory staff positions, facilities and equipment, calibration procedures, sample handling and custody procedures, contamination control and test report format and procedures;

_____ c. specific records (or reference to records) of calibration tests, samples received and their locations, contamination tests and test reports that have been issued by the laboratory; and

_____ d. schedules (for routine timing and frequency) planned for calibration, contamination monitoring and determination of the precision and accuracy of the analysts.

_____ 2.2 The laboratory's quality assurance analyses (including non-AHERA analyses) represent at least 10% of the total number of TEM asbestos analyses performed.

NOTE: *The value of 10% is a minimum value that applies to a laboratory that has: 1) trained analysts, 2) its laboratory calibrations, contamination checks and other quality systems components statistically characterized and in a state-of-control and 3) a high frequency of analyses. For laboratories not fitting these criteria, the quality assurance analyses must be a higher percentage of the total number of TEM asbestos analyses performed.*

_____ 2.3 Quality assurance analyses are performed regularly covering all time periods, sample types, instruments, tasks, and personnel. The selection of samples is semirandom and, when possible, the checks on personnel performance are executed without their prior knowledge. A disproportionate number of analyses are not performed prior to internal or external audits. Quality assurance analyses are not postponed during periods of heavy work loads.

2.4 The laboratory summarizes all of the quality assurance activities each month including:

_____ a. contamination checks, problems and corrective measures;
_____ b. accuracy of each analyst and of the laboratory;
_____ c. interlaboratory and intermicroscope analyses;
_____ d. sampling precision determined by filter reanalysis;
_____ e. calibrations;
_____ f. identification of any sample custody errors, such as mixing up samples, losing samples, etc.; and
_____ g. deficiency corrections.

2.5 The laboratory has the following documents available for reference:

_____ a. NIST Handbook 150, *NVLAP Procedures and General Requirements;*
_____ b. NIST Handbook 150-13, *NVLAP Airborne Asbestos Analysis;*
_____ c. the Environmental Protection Agency's, *Interim Transmission Electron Microscopy Analytical Methods—Mandatory and Nonmandatory—and Mandatory Section to Determine Completion of Response Actions*, Appendix A to Subpart E, 40 CFR part 763, October 30, 1987, or the current U.S. Environmental Protection Agency AEM method for the determination of completion of response actions;
_____ d. *Asbestos-Containing Materials in Schools; Final Rule and Notice*, 40 CFR, Part 763, Subpart E;
_____ e. general references on analytical electron microscopy, transmission electron microscopy, asbestos analysis, and crystallography;
_____ f. AEM manufacturer's operation manual; and
_____ g. multichannel analyzer manufacturer's operation manual.

2.6 The laboratory has references available and shall be knowledgeable on the following topics; however, the exact reference is not required:

_____ a. **for verified asbestos analysis**, see:
- E. B. Steel and J. A. Small, *Accuracy of Transmission Electron Microscopy for the Analysis of Asbestos in Ambient Environments*, Analytical Chemistry, Vol. 57, 1985, pp. 209-213;
- S. Turner and E. B. Steel, *Analysis of Transmission Electron Microscopy Analysis of Asbestos on Filters: Interlaboratory Study*, Analytical Chemistry, Vol. 63, 1991, pp. 868-872; and
- S. Turner and E. B. Steel, NISTIR 5351, *Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0*, 1994;

_____ b. **for spot size measurement**, see:
- D. B. Williams, *Practical Analytical Electron Microscopy in Materials Science*, Philips Electronics Instruments, Inc., Mahwah, New Jersey, 1984, pp. 34-35 (for TEM or STEM mode);
- D. B. Williams, *Standardized Definitions of X-ray Analysis Performance Criteria in the AEM*, in A. D. Romig Jr. and W. F. Chambers, (ed.), *Microbeam Analysis 1986*, San Francisco Press, San Francisco, 1986, pp. 443-448 (for TEM mode); and
- J. I. Goldstein, et al., *Scanning Electron Microscopy and X-ray Microanalysis*, Plenum Press, New York, 1981, p. 48 (for STEM mode);

_____ c. **for k-factor measurement**, see:
- D. C. Joy, A. D. Romig, J. I. Goldstein, *Introduction to Analytical Electron Microscopy*, Plenum Press, New York, 1986; or
- D. B. Williams, *Practical Analytical Electron Microscopy in Materials Science*, Philips Electronics Instruments, Inc., Mahwah, New Jersey, 1984;

_____ d. **for quality assurance**, see J. K. Taylor, *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, Michigan, 1987;

_____ e. **for statistical analysis**, see M. G. Natrella, *Experimental Statistics*, John Wiley & Sons, New York, 1966;

_____ f. **for control charts**, see *Manual on Presentation of Data and Control Chart Analysis*, ASTM, Philadelphia, 1991; and

_____ g. reference data on the crystallography and chemical composition of minerals that analytically interfere with the regulated asbestos minerals.

3 Personnel

_____ 3.1 Staff members are aware of both the extent and limitation of their area of responsibility.

_____ 3.2 The laboratory has a written description of its training program which includes training with standards and blind testing to determine competency and criteria for successful completion.

_____ 3.3 Analysts and technical supervisors participate in an appropriate form of continuing education, such as formal coursework, in-house education, and scientific or technical meetings, and have access to journals that describe advances in the field of electron microscopy and/or asbestos analysis.

_____ 3.4 The technical supervisor(s) shall be qualified to conduct AEM studies, apply AEM to crystalline materials and is knowledgeable in the field of asbestos analysis including procedures for sample handling, preparation, analysis, storage, disposal, and contamination monitoring.

3.5 AEM analysts are trained and are proficient in:

_____ a. AEM use, calibration, alignment, electron micrography (or functional equivalent);

_____ b. EDXA, x-ray collection and interpretation including recognition of artifacts and abnormal features in spectra resulting from detector problems, contamination or detector-sample geometry;

_____ c. electron diffraction measurement and interpretation including the determination of d-spacings, Miller indices, zone axes;

_____ d. asbestos counting methods including:

- _____ counting rules for simple and complex structures;
- _____ grid and grid square selection (nonadjacent, random);
- _____ x-y stage translation and parallel traverses;
- _____ stage positioning and repositioning;

_____ e. asbestos identification including:

- _____ morphology criteria;
- _____ crystallographic criteria through electron diffraction analysis;
- _____ chemical composition criteria through EDXA;

_____ f. differentiation between regulated asbestos minerals and other minerals that resemble the regulated asbestos minerals;

_____ g. determination of the concentration of fibers on a filter sample;

_____ h. verified asbestos analysis;

_____ i. recognition of acceptable and unacceptable sample preparations; and

_____ j. recognition of sample and instrumental artifacts.

3.6 The accuracy of asbestos identification and counting of each AEM analyst is evaluated by:

_____ a. analyses of reference materials prepared in-house and purchased;

_____ b. analyses of NIST proficiency testing samples; and

_____ c. verified analyses.

NOTE: *Additional quality assurance information may be gained by determining the precision of AEM analysts by repeat analysis of grid squares.*

3.7 Verified asbestos analyses are performed routinely by the laboratory with sufficient frequency and on sufficient types of samples to determine each operator's initial and continuing performance that the following conditions are satisfied:

_____ a. samples having approximately 6-40 structures/grid opening are used to achieve statistically significant information on new analysts;

_____ b. after initial training, a variety of asbestos loadings, including routine AHERA samples, are used to validate analysts' results. Samples include loadings seen in typical AHERA samples up to 6-40 structures/grid opening. At least 20% of verified analyses are performed on samples with 6-40 structures/grid opening loadings and at least 5 grid openings with 6-40 structures/grid opening are counted annually;

_____ c. filter blanks, unless known to be contaminated, are not used for verified counting;

_____ d. during training, all counts used in reports are verified until verified status is attained; and

_____ e. after verified status is attained, the frequency of verified analysis is at least 1 per 100 grid opening analyses.

NOTE: *Labs may find it advantageous to use up to 4 operators on one analysis to characterize initial operator performance, if an analyst with verified status is not available. An analyst that obtains verified status has an average accuracy ≥80% of true positives, ≤20% false negatives, and ≤10% false positives on both standard and field samples.*

_____ 3.8 The laboratory has a person responsible for tracking and storing samples (a laboratory sample coordinator).

_____ 3.9 The laboratory is organized so that staff members are not subject to undue pressure or inducement that might influence their judgment or results of their work. The laboratory is able to demonstrate that the sample work load required for each analyst is consistent with accurate and precise analytical measurement.

4 Accommodation (facilities) and environment

4.1 The following facilities are available:

_____ a. clean room or clean areas for sample preparation and handling separate from bulk asbestos;

_____ b. electron microscopy facility; and

_____ c. room or area for filter and grid storage separate from bulk asbestos.

4.2 The following are available in the clean room or clean area:

_____ a. class 100 (or cleaner) HEPA-filtered air under positive pressure; and

_____ b. exhaust hood for safe use of filter dissolution reagents.

4.3 Safe working conditions are maintained, including:

_____ a. safe handling of asbestos; and

_____ b. safe handling and storage of filter-dissolving reagents such as chloroform, dimethyl formamide, acetone, acetic acid, etc.

4.4 Quality system documentation contains:

_____ a. procedures for the prevention, monitoring, and control of contamination of filters and grids; and

_____ b. procedure (or flow chart) for the systematic checking for possible sources of contamination if contamination is detected. This includes checking all areas, instrumentation and materials used in the preparation and analysis of air filter samples.

4.5 To minimize the possibility of contamination of samples, the following is performed:

_____ a. all personnel are instructed in contamination prevention;

_____ b. personnel, instrumentation and materials used for the preparation of bulk materials that potentially contain asbestos are kept separate from areas used for air filter preparation, handling or analysis; and

_____ c. all reagents are checked for asbestos contamination prior to use in sample preparation.

NOTE: *Personnel who have worked with bulk samples should not subsequently be allowed to work with air filter samples on the same day. It is acceptable, however, for personnel to work on bulk samples after having worked with air samples.*

NOTE: *The next four items concern the use of blank materials for contamination monitoring. Definitions of filter lot, sealed, field and laboratory blanks are given in Section 285.5 of NIST Handbook 150-13. Other blanks should be used as needed to determine and correct sources of contamination, including blanks for AEM specimen holders, evaporators, Jaffe wick, low temperature asher, laboratory air samples, etc.*

_____ 4.6 A laboratory blank filter is present in the clean room or clean area during sample preparation and after cleaning or servicing of the clean room or clean area. The laboratory blanks are obtained from a filter lot that has been shown not to be contaminated.

4.7 The maximum allowed contamination levels for filter lot, sealed, field and laboratory blanks are:

_____ a. a cumulative average level of 18 structures per mm^2; and

_____ b. a single preparation level of 53 structures per mm^2.

4.8 Preparation of nominally blank filters is done:

_____ a. on a minimum of one laboratory blank per sample set or 10% of samples (whichever is greater);

_____ b. of a laboratory blank after cleaning or servicing the clean room or clean area; and

_____ c. on all field and sealed blanks with each series of samples (if these blanks are not identified and known to laboratory, all filter samples are prepared with the series).

NOTE: *The laboratory must properly record and archive prepared grids (even if not analyzed).*

4.9 Analysis of nominally blank filters is done:

_____ a. on a minimum of one laboratory blank per 25 filter analyses;

_____ b. of the laboratory blank when the average count for the full set of filters exceeds 70 structures/mm^2; and

_____ c. on the field and sealed blank when the full indoor/outdoor analysis is performed.

NOTE: *If the Z-test is performed by the laboratory, then the field and sealed blanks must be known to the laboratory. The laboratory is responsible for the analysis of the filter lot blanks only when contracted to analyze them by the sampling organization.*

The laboratory blank analyses can be counted towards the required 10% quality assurance analyses. The field and sealed blank analyses, however, cannot be counted towards this requirement.

_____ 4.10 When contamination above acceptable levels is found, analyses for AHERA clearance are discontinued until the cause is found and corrected or until data shows that the contamination problem no longer exists.

5 Equipment and reference materials

5.1 The following sample preparation equipment or equivalent is available in the clean room or area:

_____ a. condensation washer and/or Jaffe wick with the appropriate reagents and supplies;
_____ b. filter preparation materials (e.g., scalpel, microscope slides, tweezers, etc.);
_____ c. indexed 200-mesh TEM grids—also referred to as finder grids (only grids with uniquely identifiable grid openings may be used; grids with only a symmetrical central marking do ***not*** qualify as finder grids); and
_____ d. other materials as needed.

5.2 The laboratory has a low temperature plasma asher which:

_____ a. is supplied with oxygen;
_____ b. allows for control of speed of evacuation and venting to minimize disturbance of particles on filter surface; and
_____ c. is not used for bulk samples (asbestos or other).

5.3 The laboratory has:

_____ a. a carbon evaporator which attains a vacuum of 13 Pa (10^{-4} torr) or lower and has controlled venting to atmospheric pressure;
_____ b. spectrochemically pure carbon rods;
_____ c. a carbon rod sharpener; and
_____ d. gold or aluminum wire for evaporation (or have sputter coater with gold target).

5.4 The laboratory has an electron microscope, which has the following under routine asbestos analysis conditions:

_____ a. capability of operation at a voltage between 80 keV-120 keV;
_____ b. capability of producing an electron diffraction pattern of a single fibril of chrysotile;
_____ c. capability of displaying and resolving hollow tube of chrysotile;
_____ d. capability of precise fiber length (at 0.5 μm and 5.0 μm) and diffraction pattern measurement, regardless of image (fiber or pattern) orientation (often fulfilled through use of a fluorescent screen with calibrated gradations in the form of circles or at least two perpendicular lines);
_____ e. mechanical stage with linear, reproducible movements along two perpendicular directions;
_____ f. capability of producing a spot at crossover that is $\leq$ 250 nm during EDXA analysis; and
_____ g. an imaging system for recording brightfield images and electron diffraction patterns on electron micrographs or on other suitable media.

NOTE: *It is strongly recommended that the laboratory possess a holder capable of obtaining zone axis diffraction patterns (either a double-tilt or rotation-tilt holder).*

5.5 The laboratory is able to record and produce hard copies of images (on electron micrographs or other media) to document:

_____ a. visibility of chrysotile hollow tubes and beam damage;
_____ b. visibility and measurement of electron diffraction patterns, in particular chrysotile (002), (004), (110), (020), (130), and (200) reflections;
_____ c. complex arrangement of fibers;
_____ d. a range of magnifications from 1000 × to 100 000 × in brightfield imaging mode; and
_____ e. a range of diffraction camera lengths that enable accurate diffraction pattern measurement (approximately 20 cm to 80 cm).

_____ 5.6 An EDXA system is interfaced to all electron microscopes used for asbestos analysis.

5.7 The EDXA system (detector and multichannel analyzer), under routine analysis conditions, meets the following specifications:

_____ a. 175 eV or better resolution at Mn Kα peak;
_____ b. proven detection of Na peak in standard crocidolite or equivalent;
_____ c. capable of obtaining statistically significant Mg and Si peaks from a single fibril of chrysotile; and
_____ d. consistent relative sensitivity factors over large areas of the specimen grid.

NOTE: *(1) A low background holder may be necessary to meet these requirements, (2) for item 5.7b, the Na K-lines and Cu L-lines (potentially from the Cu TEM grid) have significant overlap and care must be taken to show that Na is measured above the Cu L-line background.*

5.8 The multichannel analyzer has the following:

_____ a. software capable of obtaining background corrected peak intensities or integrals for Na, Mg, Al, Si, Ca, Fe and other elements as needed;
_____ b. capability of accumulation and display of an x-ray spectrum (minimum 0.7 keV-10 keV); and
_____ c. capability of making a hard copy of an x-ray spectrum.

5.9 The following standards and any associated certificates are available:

_____ a. materials with a certified value for the loading of asbestos on filters
  _____ NIST SRM 1876b
  _____ optional - NIST RM 8410 and RM 8411
_____ b. materials that are characterized as asbestos for training and analyst evaluation;
  _____ NIST SRMs 1866 and 1867 or NIST-traceable standard
_____ c. calibration material(s) for the x-ray system
  _____ SRM 2063 or NIST-traceable standard;

_____ d. standard optical grating replica for magnification calibration; and
_____ e. gold or aluminum film material for electron diffraction calibration.

NOTE: *(1) The laboratory must use SRM 1876b for calibration and not SRMs 1876 or 1876a. SRM 1876 and 1876a were certified using sets of counting rules that are no longer in use. (2) SRMs 1866 and 1867 contain bulk asbestos and, therefore, precautions need to be taken against contaminating the filter preparation area and AEM with these specimens. (3) The laboratory has the primary responsibility for developing or obtaining a set of standards useful for checking the identification, analysis and concentration of asbestos on filters. For example, internal standards can be drawn from samples received by the laboratory or developed by the laboratory through water filtration of asbestos mixtures or by other methods. The samples then must be well-characterized by the laboratory for use as standards. NVLAP proficiency testing samples do not qualify as NIST-traceable standards.*

6 Measurement traceability and calibration

NOTE: *Control charts should be constructed to show calibrated values vs. time, the magnitude of their variation, and the allowable limits of variation. The magnitude of variation specified for many calibrations in this program is defined as 2 s (s is the estimated standard deviation of a set of measurements). Initially, many (15-30) calibrations should be performed in a few month's time to establish a baseline for variation in the measurement. If the variation is within specified limits and the accuracy is acceptable, the frequency of the calibration can be reduced. In general, the majority of calibrations should have been done within 3 months prior to analyses performed for clients.*

All calibrations should be performed with the instrument, stage, sample, x-ray detector and other parameters at routine asbestos analysis conditions (e.g., tilt, apertures, location, specimen height, accelerating voltage, etc.) and with the microscope aligned. Tilting the viewing screen and specimen grid during fiber measurement, or the viewing screen during diffraction measurement is not recommended. Laboratories using tilts must demonstrate the required measurement accuracy and precision for all fiber and diffraction maxima orientations.

_____ 6.1 The laboratory has specific procedures in its quality system documentation for the development and use of control charts.

_____ 6.2 The laboratory uses control charts to summarize calibration data.

6.3 The magnification of the electron microscope is calibrated:
_____ a. using an optical diffraction grating replica (the variation in the calibration measurements (2 *s*) is $<5\%$ of the mean calibration value);
_____ b. for magnifications commonly used for asbestos analysis and for any other magnification used for measurement (e.g., the magnification used to measure grid square size); and
_____ c. on all measurement systems applied in the laboratory for asbestos analysis such as the phosphor viewing screen, film, monitor and/or image analysis system.

6.4 The accuracy and precision of measurements at 0.5 μm are determined by:

_____ a. calibration of the measuring system(s) (on screen, film, monitor, and/or image analysis system) at 0.5 μm; and

_____ b. repeat analysis by the same and different analysts of asbestos fibers approximately 0.5 μm in length. (This data may be derived in part from verified analysis data for fibers close to 0.5 micrometers in length.)

6.5 The diffraction camera constant is calibrated:

_____ a. using an evaporated gold or aluminum film (the variation in the calibration measurements (2 *s*) is $<5\%$ of the mean calibration value);

_____ b. for the camera lengths commonly used for asbestos analysis;

_____ c. under the conditions used for asbestos analysis; and

_____ d. on all measurement systems including the TEM screen, film, monitor, image analysis system and/or any other system as applied in the laboratory for asbestos analysis.

NOTE: *A minimum of three measurements at 45-degree angles are required on the innermost ring. These measurements will allow detection of deviations of a ring from a circle. Measurements of at least two of the outer rings must be made to monitor for radial distortions and to ensure that an error in measurement of the inner ring did not occur. If significant distortion is found, more measurements are needed for better characterization.*

_____ 6.6 The beam dose is calibrated so that beam damage to chrysotile is minimized—specifically so that an electron diffraction pattern from a single fibril $\geq$ 1 μm in length from a NIST SRM chrysotile sample is stable in the electron beam for at least 15 seconds.

_____ 6.7 The laboratory has recorded the setting of the electron microscope (condenser aperture, spot size, etc.) that allows for the stability of chrysotile specified in item 6.6 above. This setting is used as the standard operation procedure for routine analyses of possible chrysotile structures.

6.8 The spot size of the electron beam used for well resolved x-ray microanalysis is determined and the:

_____ a. the average spot size for a properly stigmated beam is $\leq$ 250 nm; and

_____ b. the variation in diameter measurements (2 *s*) is $<$ 25% of the mean value.

6.9 The EDXA system is shown through calibration data to have:

_____ a. a resolution (full-width, half-maximum) for Mn Kα that is $<$ 175 eV; and

_____ b. a value for the sum of the resolution and the variation (2 s) that is $<$ 180 eV.

_____ 6.10 The x-ray energy vs. channel number for the EDXA system is calibrated to within 20 eV for at least two peaks between 0.7 keV and 10 keV. One peak

should be from the low end (0.7 keV to 2 keV) and the other peak from the high end (7 keV to 10 keV) of this range. The calibration of the x-ray energy is checked prior to each analysis of samples and recalibrated if out of the specified range.

6.11 The relative sensitivity (k-factors) factors relative to Si for elements found in asbestos (Na, Mg, Al, Si, Ca, Fe) are determined so that:

_____ a. the k-factors are determined to a precision (2 *s*) within 10% relative to the mean value obtained for Mg, Al, Si, Fe, and within 20% relative to the mean value obtained for Na;

_____ b. the k-factor relative to Si for Na is between 1.0 and 4.0, for Mg and Fe is between 1.0 and 2.0, and for Al and Ca is between 1.0 and 1.75; and

_____ c. the k-factor for Mg relative to Fe on SRM 2063(a) or other standard traceable to NIST is 1.5 or less.

NOTE: *SRM 2063 or SRM 2063a can be used for the determination of k-factors for Mg, Si, Ca and Fe. The laboratory must obtain its own chemically characterized materials for determining the Na and Al k-factors. Examples include albite for Na k-factor determination and biotite or albite for Al k-factor determination. Na k-factors are sensitive to electron beam dose (current and time). It is suggested that small particles (≤ 0.1 μm in size) be used for Na k-factor determination to minimize the effect of Na migration.*

_____ 6.12 The portions of a grid in a specimen holder for which abnormal x-ray spectra are generated under routine asbestos analysis conditions are determined and these areas are avoided in asbestos analysis.

NOTE: *X-rays can be absorbed due to the relative position of the area of interest, the grid bars, specimen holder and x-ray detector and give an abnormal spectra (for an example of an abnormal spectra see S. Turner, E. B. Steel, S. S. Doorn, and S. B. Burris, "Proficiency Tests for the NIST Airborne Asbestos Program - 1991," NISTIR 5432). The laboratory should use a standard material (SRM 2063 is recommended) to map out the spectra obtained over the grid area and to thereby determine the regions that should be avoided in routine analysis.*

_____ 6.13 The low temperature asher is calibrated by determining a calibration curve for the weight vs. ashing time of collapsed mixed-cellulose-ester (mce) filters.

NOTE: *The AHERA method specifies that a mixed-cellulose-ester filter is to be ashed by 10%. However, if ashing by this amount generates a texture in the replica that affects structure counting, it is permissible to etch by less than this amount.*

_____ 6.14 The determination of the quality of sample preparations is calibrated or the laboratory has the following documentation available:

_____ a. images and samples showing good preparations and examples of the types of problems that occur in poor preparations (readily available to analysts); and

_____ b. a record of repeat evaluations of images and samples by the same and different analysts. (This data may be derived in part from sample preparation evaluations done in the course of verified analysis.)

_____ 6.15 The magnification of the grid opening measurement system is calibrated using an appropriate standard. The variation in the calibration measurements (2 *s*) is $<5\%$ of the mean calibration value.

_____ 6.16 Trained AEM analysts have an average accuracy $\geq 80\%$ of true positives, $\leq 20\%$ false negatives, and $\leq 10\%$ false positives (these data are reported on a structures per grid square basis).

_____ 6.17 The laboratory and AEM analysts obtain mean analytical results on SRM 1876b so that trimmed mean values fall within 80% of the lower limit and 110% of the upper limit of the 95% confidence limits as published on the certificate (these limits are derived from the allowable false positives and false negatives given in the previous item). The SRM is analyzed a minimum of once a year by each AEM analyst.

_____ 6.18 The laboratory has documentation demonstrating that AEM analysts correctly classify at least 90% of both bundles and single fibrils of asbestos structures $\geq$ 1 μm in length in known standard materials traceable to NIST (such as the bulk asbestos SRM 1866).

_____ 6.19 Interlaboratory analyses are performed to detect laboratory bias. The frequency of interlaboratory verified analyses corresponds to a minimum of 1 of 200 grid square analyses for clients.

_____ 6.20 If more than one AEM is used for asbestos analysis, intermicroscope analyses are performed to detect instrument bias.

_____ 6.21 The sampling precision is determined by repeat preparation and analysis of the same filter by the same and different analysts.

_____ 6.22 Required calibrations are performed correctly and on a frequent enough basis to ensure accurate results.

7 Test methods and calibration

_____ 7.1 The laboratory uses Environmental Protection Agency, "Interim Transmission Electron Microscopy Analytical Methods—Mandatory and Nonmandatory—and Mandatory Section to Determine Completion of Response Actions," Appendix A to Subpart E, 40 CFR part 763, October 30, 1987, and any NIST or U.S. Environmental Protection Agency clarifications, modifications, or updates to the TEM method for the analysis of asbestos.

_____ 7.2 Quality system documentation details the AEM method as it is applied in the laboratory. (A simple copy of the AHERA method is not sufficient). If departures are made from the method, the laboratory has written procedures detailing how the analyses are conducted.

7.3 The laboratory has written procedures for:

_____ a. preparation of mixed-cellulose-ester filters, including techniques for collapsing, etching, carbon coating and dissolution of filters;

_____ b. preparation of polycarbonate filters, including techniques for carbon coating and dissolution of filters; and

_____ c. determination of the number of grid squares and grid area to be analyzed per sample.

7.4 Laboratory personnel:

_____ a. prepare at least three grids per filter; and

_____ b. analyze approximately half of the predetermined sample area to be analyzed on one grid and the remaining half on a second grid preparation.

7.5 The laboratory has written procedures for the evaluation of the quality of prepared grids. The criteria for acceptance include:

_____ a. the percentage of grid openings covered by the replica section (coherent or noncoherent) is greater than approximately 50%;

_____ b. the percentage of grid openings covered by the replica section that:

_____ are intact is greater than approximately 50%;

_____ have undissolved filter is less than approximately 50%; and

_____ have overlapping or folded replica is less than approximately 50%.

_____ c. at least 20 grid squares have no overlapping or folded replica, < 5% holes and < 5% opaque area due to incomplete filter dissolution. "Opaque area" means that the sample preparation artifact is sufficiently opaque to the electron beam that recognition and analysis of fibers will be difficult or impossible.

NOTE: *NIST Interagency Reports, in preparation, will give a description of sample preparation features and a procedure for choosing grid squares for analysis.*

_____ 7.6 The laboratory has written procedures for the determination of the area of grid squares.

NOTE: *The AHERA method requires that either 1) the area of the grid square analyzed be determined, or 2) the average area of grid squares in a grid lot of 1000 be determined by measurement of 20 grid squares on each of 20 grids. If premeasured grids are purchased, the laboratory should confirm the measurements as a quality assurance procedure. Initially, many grid squares should be remeasured. If the values are in agreement with those given with the grids, then the number of remeasurements can be reduced to approximately 5% of those required by the AHERA method. Premeasured grids must have a report of analysis which gives the mean grid opening area, the number of grids and openings measured, the standard deviation of the opening area, and the method of analysis.*

7.7 The laboratory has written procedures for operation of the AEM for asbestos analysis including:

_____ a. method for alignment of the electron microscope so that the electron beam travels down the optic center of the column. This includes alignment of the electron gun, apertures, and tilt as described in the manufacturer's and laboratory's operating manual; and

_____ b. standard operating conditions of the AEM

_____ voltage (between 80 keV-120 keV)

_____ microscope magnification (15 000× - 20 000× for analysis).

_____ 7.8 There is documentation to show that the quality of alignment of the electron microscope is checked daily or prior to each use for analyses and calibrations. The alignment is checked by, at minimum, changing the magnification, spot size, and image focus and by checking the stigmation of the electron beam. The AEM analyst aligns the electron microscope if the instrument does not meet the laboratory's alignment criteria as stated in the quality manual. (Note: Alternatively, the AEM can be aligned daily or prior to each use).

7.9 The laboratory has written procedures for examining a grid square and for counting and analyzing particles (a detailed description is necessary—a copy of the EPA method is not sufficient) including:

_____ a. method for recording grid orientation in the microscope;

_____ b. particle loading acceptance criteria (>10% by area particulate loading or uneven particle loading is rejected);

_____ c. unique grid and grid square labelling system (indexed grids);

_____ d. grid square traversing method, including the use of orthogonal scans;

NOTE: *The intent is to completely cover the grid square without having structures missed or counted twice. To do this, parallel, overlapping traverses are made across a grid square. Care is taken to move only one translator during a traverse. If an asbestos structure is encountered and the other translator is moved for analysis, then the stage is returned to the original traverse position before continuation of the traverse.*

_____ e. recording rules;

_____ f. structure counting rules; and

_____ g. determination of whether a sample set passes or fails AHERA clearance if required by the client.

7.10 The laboratory has quality system documentation which contains criteria for:

_____ a. identification of electron diffraction patterns of regulated asbestos minerals and of nonasbestos minerals, including those that closely resemble regulated asbestos minerals;

_____ b. identification of EDXA spectra of regulated asbestos minerals and of nonasbestos patterns, including those that closely resemble regulated asbestos minerals; and

_____ c. differentiating asbestos minerals from at least the following phases: the pyroxenes, hornblende, wollastonite, halloysite, palygorskite,

sepiolite, antigorite, lizardite, talc, and vermiculite. The minimum criteria for differentiation must be presented.

_____ 7.11 AEM analysts record sufficient information for each analysis so that a verified analysis can subsequently be performed.

NOTE: *Information sufficient for performing a verified analysis includes the orientation of the grid at the analysis magnification, a sketch (or image) for each structure and the size of each structure (the recording of the location of the structure is also of use). Recording this information will allow for random quality assurance checks of any analysis and removes the bias that can occur when verification is done with the analysts' foreknowledge. The laboratory may want to refer to E. S. Windsor, S. Turner and E. B. Steel, NISTIR 5358, in which a recording form suitable for verification is described.*

_____ 7.12 AEM analysts record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. The identification of diffraction patterns is verified by a qualified individual. It is shown that the AEM analyst is correct 80% of the time in identification of recorded diffraction patterns.

_____ 7.13 The laboratory has written procedures for verifying report calculations.

8 Handling of calibration and test items

8.1 The log-in system includes documentation of:

_____ a. the date of receipt;
_____ b. identity of the client;
_____ c. unique identification for sample;
_____ d. air volume pulled through sample;
_____ e. filter pore size;
_____ f. condition of the samples; and
_____ g. acceptance or rejection of the samples.

_____ 8.2 The laboratory has written criteria for acceptance or rejection of filter cassettes.

NOTE: *Examples of rejection criteria include: insufficient sampling documentation, bulk samples included with air filter samples, filter cassettes open, filters overloaded with particulate, uneven particle loading, sampling parameters not meeting AHERA sampling criteria, filters not uniquely identified, filters of incorrect pore size, tampering with cassettes evident, sample that laboratory is not capable of preparing properly, etc.*

8.3 The laboratory has a documented chain-of-custody system by which the following is recorded:

_____ a. location of sample;
_____ b. a listing of personnel that have handled or worked with the sample; and
_____ c. a listing of what has been done to the sample.

8.4 The laboratory:

_____ a. stores the unused portions of filters in their cassettes for at least 30 days;

_____ b. stores all prepared grids (even if not analyzed) for at least three years; and

_____ c. stores the filters and grids in a logical fashion so that specified samples can be retrieved within one working day.

9 Records

_____ 9.1 All records are retained for a minimum of three years and are stored in a logical fashion allowing retrieval within one working day.

_____ 9.2 The laboratory has documentation, either electronic backup or "paper" hard copy, to ensure survival of original data if computers are used for data retention.

9.3 The quality system documentation contains standardized methods (forms) for recording the following:

_____ a. log-in of samples;

_____ b. criteria for acceptance or rejection;

_____ c. evaluation of quality of prepared grids; and

_____ d. AEM sample analysis data.

9.4 The laboratory has records relating to:

_____ a. sample custody; and

_____ b. contamination monitoring.

9.5 Records related to contamination include results and timing of all checks of the following:

_____ a. filter lot blanks;

_____ b. field blanks;

_____ c. laboratory blanks;

_____ d. all other areas and samples, as needed, to track contamination;

_____ e. summary of contamination problems and resolution; and

_____ f. a summary of blank results in control chart or similar format.

9.6 All records related to quality assurance testing of staff and laboratory are retained including results of:

_____ a. analyses of reference materials;

_____ b. analysis of NIST proficiency testing materials;

_____ c. verified analyses;

_____ d. interlaboratory analyses;

_____ e. intermicroscope analyses (if the laboratory uses more than one AEM for asbestos analysis);

_____ f. repeat preparation and analysis of same filter by same analyst and by different analysts;

_____ g. identification of mineral types; and

_____ h. evaluation of filter preparations.

9.7 Records related to the AEM analysis of a filter include:

_____ a. general information:
- _____ operator (analyst must sign and date analysis sheet),
- _____ sample identification,
- _____ client identification,
- _____ date;

_____ b. instrument used (if more than one available);

_____ c. operating parameters of the instrument used including:
- _____ magnification,
- _____ accelerating voltage,
- _____ other, as needed to ensure alignment and calibration compliance;

_____ d. filter and grid related information:
- _____ filter sampling data sheet as received with sample,
- _____ filter type,
- _____ area of grid squares analyzed,
- _____ number of grids prepared and their location,
- _____ evaluation of prepared grids,
- _____ orientation of grid in AEM,
- _____ grids and grid squares analyzed;

_____ e. original data records include (for AHERA analysis):
- _____ structure type (fiber, bundle, cluster, matrix),
- _____ the number of fibers that are $\geq$ 0.5 micrometers and $<$ 5 micrometers,
- _____ the number of fibers that are $\geq$ 5 micrometers,
- _____ classification of structures as chrysotile, amphibole (as grunerite (Amosite), riebeckite (crocidolite), anthophyllite, actinolite, or tremolite), or nonasbestos,
- _____ at a minimum, measurement results of both EDXA and electron diffraction for each structure (usually the first 4) identified as amphibole that caused the concentration of regulated asbestos minerals on the filter to reach or exceed 70 structures/mm^2,
- _____ at a minimum, measurement results of electron diffraction for each structure (usually the first four) identified as chrysotile, that caused the concentration of regulated asbestos minerals on the filter to reach or exceed 70 structures/mm^2,
- _____ documentation of positive electron diffraction ***or*** EDXA for each chrysotile asbestos structure subsequent to the asbestos structure that caused the concentration on the filter to reach or exceed 70 structures/mm^2, and documentation of positive EDXA or measured zone axis diffraction pattern, for each amphibole structure subsequent to the asbestos structure that caused the concentration of asbestos on the filter to reach or exceed 70 structures/mm^2,
- _____ at a minimum, documentation or measurement of results of EDXA and/or measurement of a zone axis electron

diffraction pattern for each structure in the nonasbestos class that corresponds to a concentration of over 70 structures/mm^2,

_____ micrograph numbers or appropriate identification for the required one electron diffraction pattern for every five samples that contain asbestos and for any other patterns taken,

_____ criteria used to classify particles as nonasbestos, that is the property or properties that differentiate it from regulated asbestos minerals;

NOTE: *For structures whose qualitative chemical composition is distinct from regulated asbestos minerals, e.g., gypsum, only documentation of the qualitative chemical composition is necessary. For structures that have similar qualitative composition, semiquantitative measurement of composition by EDXA and/or measurement of the distinguishing features of the diffraction pattern is required.*

Measurement results include sufficient quantitative data (e.g., x-ray intensities or diffraction maxima d-spacing) to identify regulated asbestos minerals positively, as defined by laboratory identification criteria. Documentation of positive diffraction or EDXA means that the analyst records (e.g., checks off) that these properties visually and/or qualitatively match the lab's identification criteria.

_____ f. information related to report to client:

_____ concentration of asbestos in structures/mm^2 on filter and structures/cm^3 in sampled air,

_____ number of asbestos structures counted,

_____ types of asbestos,

_____ area analyzed,

_____ volume of air sampled; and

_____ 9.8 A cumulative record of results from precision and accuracy testing is maintained and summarized at least monthly.

10 Certificates and reports

10.1 Test reports include the following information for each sample set:

_____ a. area of filter analyzed;

_____ b. volume of air sampled (with reference to sampling data sheet);

_____ c. analytical sensitivity used for the analysis;

_____ d. number of total asbestos structures and number of structures by asbestos type (chrysotile, grunerite, riebeckite, anthophyllite, tremolite, or actinolite);

_____ e. concentration in asbestos structures/mm^2 of filter and asbestos structures/cm^3 of air for total asbestos structures, and with data broken down by size ($\geq$ 5 μm and $\geq$ 0.5 μm to $<$ 5 μm), and by asbestos type;

_____ f. statement of analytical uncertainty, including 95% confidence limits on the reported concentration and laboratory and analyst accuracy and precision;

NOTE: *A NISTIR containing a procedure for determining the uncertainty of measurements due to sampling is in preparation. Upon notification, the laboratories should report this uncertainty for their measured values. Subsequently, additional information will be issued regarding reporting uncertainty of the measurement.*

_____ g. micrograph number of any recorded diffraction patterns;
_____ h. copy of AEM analysis data record with analyst's signature or initials; and
_____ i. descriptions of any departures from the test method.

10.2 The following additional information shall be supplied if asbestos abatement clearance is determined to be necessary:

_____ a. calculation formulas;
_____ b. all calculation variables and constants; and
_____ c. all calculation results.

11 Subcontracting of calibration or testing

See General Operations Checklist

12 Outside support services and supplies

See General Operations Checklist

13 Complaints

See General Operations Checklist

14 Proficiency testing

14.1 The laboratory participates in mandatory airborne asbestos proficiency testing, which includes (but is not limited to) the following:

_____ a. the laboratory has written procedures for handling, analysis, and use of NIST proficiency testing materials;
_____ b. analyses are not contracted out to another laboratory;
_____ c. the laboratory keeps and uses proficiency testing materials as in-house instructional materials, unless otherwise directed;
_____ d. all analysts (full and part time) participate in all proficiency testing rounds (all analysts need not participate in proficiency testing prior to returning the results to NVLAP, but all analysts shall participate without prior knowledge of the testing results at a later date);
_____ e. each analyst separately analyzes, records and reports test results;
_____ f. a single result is reported back to NVLAP by the laboratory unless otherwise specified in the testing instructions;

_____ g. procedures and calculations (if any) are documented as to how a single result is determined;
_____ h. test results are used for interanalyst comparisons;
_____ i. corrective actions are taken and documented for problems indicated by proficiency testing;
_____ j. plans are developed and implemented for resolving problems and are documented; and
_____ k. test results, when applicable, are used in determining accuracy and precision for each analyst.

15 Subfacilities

_____ 15.1 A subfacility **is technically dependent** on the main facility (i.e., technical management and supervision are provided by the main facility).

_____ 15.2 Quality assurance activities of the subfacility are directed by the main facility.

15.3 The nature, scope, and frequency of on-site quality assurance reviews by the main facility quality manager (or equivalent) are:

_____ a. clearly defined in the quality manual; and
_____ b. appropriate for the nature and scope of work performed by the subfacility.

_____ 15.4 All permanent quality assurance and personnel records are retained at the main facility.

_____ 15.5 Quality assurance data from each subfacility are compared each month to both the main facility's data and to data from other subfacilities. Records of such comparisons are retained in quality assurance records, along with actions taken to evaluate and resolve differences.

_____ 15.6 Analysts at subfacilities participate in NVLAP proficiency testing and records are maintained of individual results.

SPECIFIC OPERATIONS CHECKLIST - COMMENTS AND DEFICIENCIES

Instructions to the Assessor: Use this sheet to document comments and deficiencies. For each, identify the appropriate item number from the checklist. Identify comments with a "C" and deficiencies with an "X." If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	*Comments and/or Deficiencies*

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